Platinum Recycling Technology Development

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Project Overview/Objectives

- To assist the DOE in demonstrating a cost effective and environmentally friendly recovery and re-use technology for PGM containing materials used in fuel cell systems.
- The initial objectives:
  - Development of lab scale processes for the solubilizing catalyst coated membranes
  - Development of Lab scale processes for the separation of catalyst and ionomer materials
  - Develop test methods to determine vitality of the recovered materials
  - Partner with the key stakeholders in this technology area

Budget

- Total Project Costs: $3.31 Million
- DOE Share: $2.65 Million
- Cost Share: $0.66 M

Technical Barriers and Targets

- DOE Technical Barriers for Fuel Cell Components
  - O. Stack Material and Manufacturing Cost:
    - **PGM costs could become unstable if fuel cell systems are introduced without the a recovery industry's ability to meet demands**
    - **Material costs of Ionomer and PGMs can be driven down during near-term commercialization, could be up to 10x reduction for ionomer**
  - P. Durability
    - New separation processes developed here will help identify failure modes of PEM fuel cell materials
- DOE Technical Target for Fuel Cell Stack System for 2010
  - Cost $35/kW
  - Durability 5000 hours

Approach

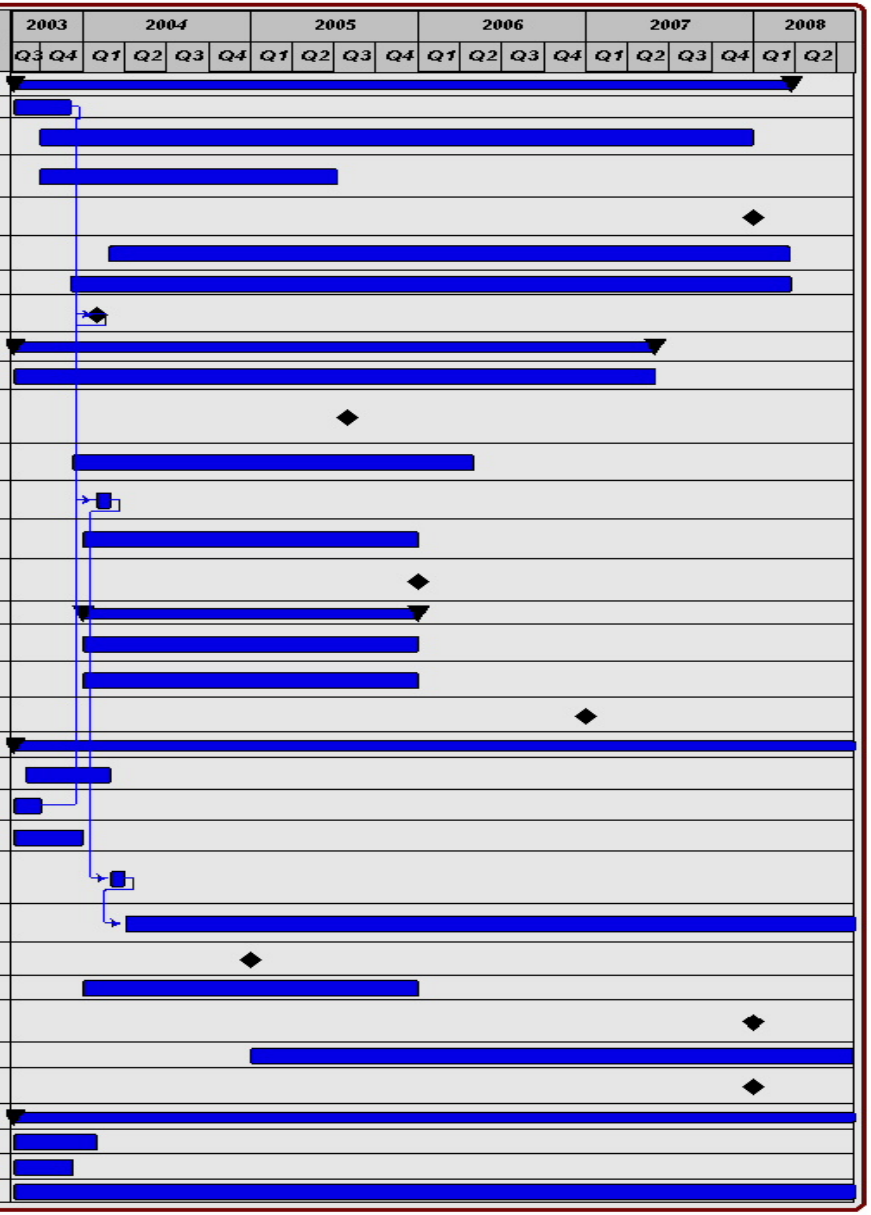
- Development of a technology that can re-manufacture spent fuel cell components into fresh fuel cell components.
  - Use pilot scale equipment that can operate on 1 sq meter (5 kW) of CCMs at one time
  - Separate ionomer and catalyst to levels needed for re-manufacture
  - Use analytical techniques to determine the differences between used and virgin materials
  - Will learn failure modes of CCM component
  - Determine the limits of separation technologies

Project Safety

- Pressure relief and over temperature alarms on all pressure vessel equipment
- Implementation of OSHA approved Chemical Hygiene Program
- Regular OSHA inspections of facilities
- H_2 Sensor with e-stop on all H_2 supplied equipment

Project Timeline

ID	Task Name	Start	End	Duration
1	Task 1 MEA Dissolution	8/7/2003	3/21/2008	241.40w
2	SubTask 1.1 Procure and install Autoclave	8/7/2003	12/5/2003	17.40w
3	Sub Task 1.2 Develop Decontamination Proceedures	10/1/2003	1/1/2008	222w
4	Sub Task 1.2.1 Develop Solution Filtration Processes	10/1/2003	7/8/2005	92.60w
5	Milestone Task 1 Identify all Significant Contaminates on EOL MEAs	1/1/2008	1/1/2008	0w
6	Sub Task 1.2.2 Study to Identify Contaminates in Used MEAs	3/1/2004	3/17/2008	211.20w
7	Sub Task 1.3 Develop Dissolution Proceedures	12/8/2003	3/21/2008	224w
8	Milestone Task 1 Demonstrate Dissolution process on MEA	2/2/2004	2/2/2004	0w
9	Task 2 Separation of NAFION(r) from Catalyst	8/7/2003	6/1/2007	199.40w
10	Sub Task 2.1 Selection of Separation Method	8/7/2003	6/1/2007	199.40w
11	Milestone Task 2 Identification of a Catalyst/ Ionomer separation process < 1% ionomer remains with catalyst	8/1/2005	8/1/2005	0w
12	Sub Task 2.1.1 Measurement of Separation Selectivity	12/12/2003	5/1/2006	124.40w
13	Sub Task 2.1.2 Perform separation of dissolved 1st batch of MEA	2/2/2004	3/3/2004	4.60w
14	Sub Task 2.3 Identification of recovered NAFION(r) properties comparison to virgin NAFION(r)	1/1/2004	12/30/2005	104.40w
15	Milestone Task 2: Aged, Recovered, Purified Ionomer shows vitality within 10% of virgin ionomer	1/2/2006	1/2/2006	0w
16	Task 3 Evaluation of Fresh and End of Life Catalysts	1/1/2004	12/30/2005	104.40w
17	SubTask 3.1 Development of Catalyst Characterization Tools	1/1/2004	12/30/2005	104.40w
18	SubTask 3.2 Evaluation and Comparison of fresh and end of life catalysts	1/1/2004	12/30/2005	104.40w
19	Milestone Task 3 Recovered Catalyst shows vitality within 10% of Virgin Catalyst	1/1/2007	1/1/2007	0w
20	Task 4 Re-Manufacture and Test of MEAs	8/7/2003	9/5/2008	265.40w
21	Task 4.1 Prepare Site Installation for aging stacks/ systems	9/1/2003	3/1/2004	26.20w
22	Taks 4.2 Manufacture 1 sq m of MEAs initial for dissolution/separation experiments	8/7/2003	10/2/2003	8.20w
23	Task 4.3 Identify stack/system partners	8/7/2003	12/31/2003	21w
24	Task 4.4 Remanufacture 1st stack worth of MEAs from separated materials recovered from fresh MEAs	3/4/2004	4/2/2004	4.40w
25	Sub Task 4.5 Ongoing real life aging of Fresh and re-manufactured MEAs	4/5/2004	9/5/2008	231w
26	Milestone Task 4 Demonstrate Worlds First Re-manufactured MEA	1/3/2005	1/3/2005	0w
27	Task 5 Vapor Phase Extraction of PGM	1/1/2004	12/30/2005	104.40w
28	Milestone Task 5: Demonstration of Reverse MOCVD Catalyst Stripping Process	1/1/2008	1/1/2008	0w
29	Task 6 Process Evaluation	1/3/2005	8/1/2008	187w
30	Milestone Task 6 Industrial Scale Cost Estimate of Re-manufacture process	1/1/2008	1/1/2008	0w
31	Task 7 Project Management	8/7/2003	9/4/2008	265.20w
32	Sub Task 7.1 Develop Subcontracts	8/7/2003	2/2/2004	25.60w
33	Sub Task 7.2 Prepare Management Plan	8/7/2003	12/11/2003	18.20w
34	Sub Task 7.3 Reporting, Quarterly	8/7/2003	9/4/2008	265.20w



Milestone Schedule

Task 1:Demonstration MEA dissolution process

Identification of all significant contaminates in EOL MEAs

Task 2: Identification of a catalyst/NAFION® separation process

Identification of EOL NAFION® vitality

Task 3: Identification of EOL Catalyst vitality

Task 4: Demonstration of world's first Re-Manufactured MEA

Task 5: Demonstration of Rev. MOCVD catalyst stripping

Task 6: Industrial Scale Cost Estimation of Process

Task 7: Reporting Requirements

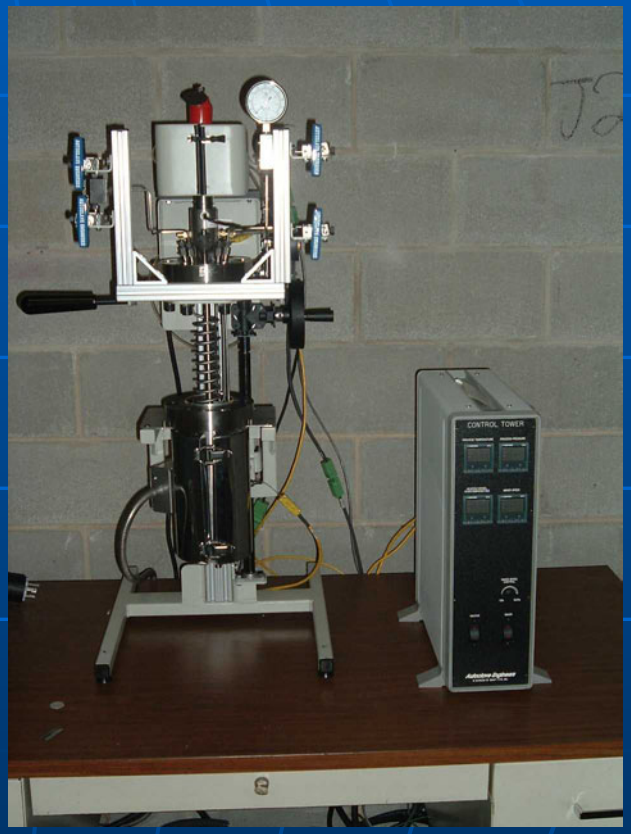
TASK 1 MEA Dissolution

-Operated on Fresh CCM, Both Ion Power and DUPONT made

-Solids Decontamination issues, remaining solids such as Kapton frame material easily separated out after run

-Many more decontamination issues will arise once we use actual aged MEAs

-Wait to use Aged MEAs until analytic techniques perfected (Task 3)



1 Liter Autoclave can process 1 sq meter of MEA at a time

TASK 2 Separation

-Centrifuge with double Rinse step still leaves some NAFION® in the catalyst powder

- -Target for Re-manufacture is ~ 50wt% NAFION®
- -Target for Recycle is ppm

-Via colormetric methods we estimate Pt/C is removed to 150 ppm from the NAFION®

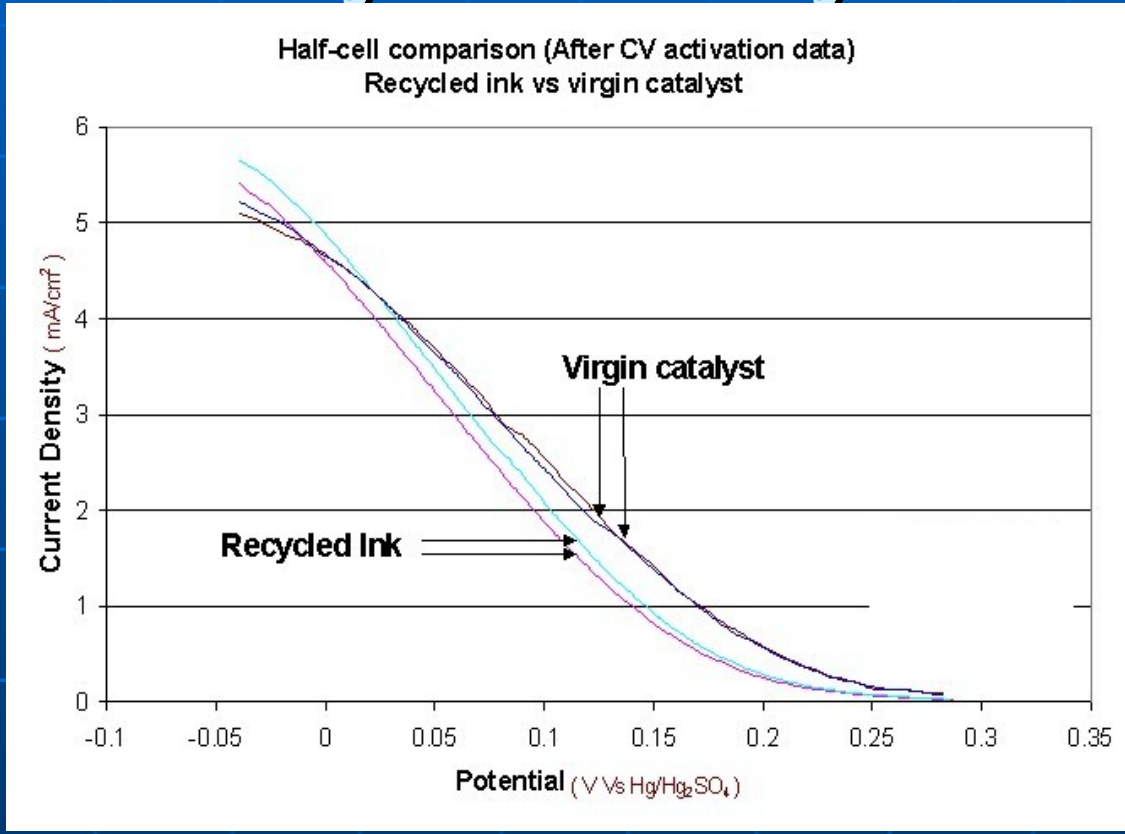
- -Target for Remanufacture may be adequate; this sample to be film cast and tested

Sorvall SUPERSPEED 20krpm Centrifuge, 8 x 30 ml

TASK 3 Catalyst Vitality

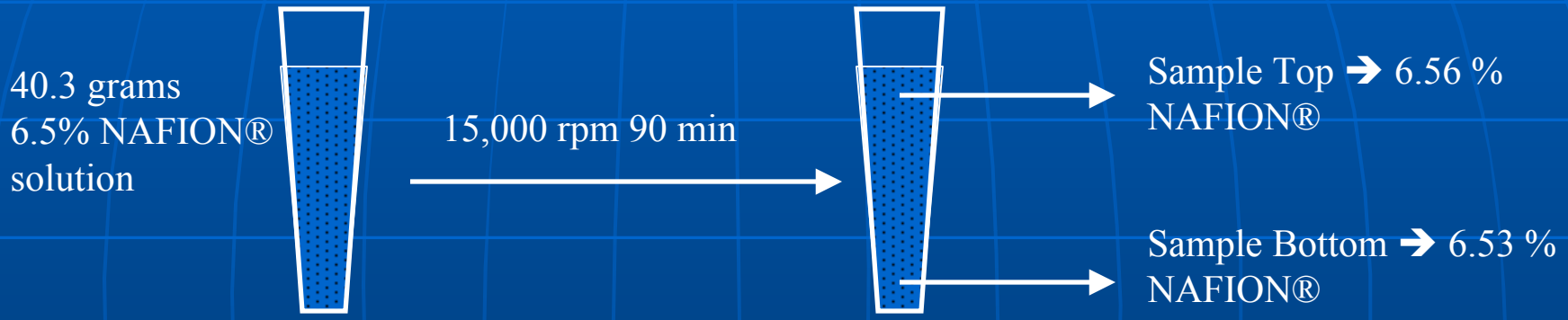
½ cell catalyst activity test on virgin versus recovered catalyst.

This ½ cell test may have some sample preparation effects that limit the activity of the recycle catalyst; still under investigation

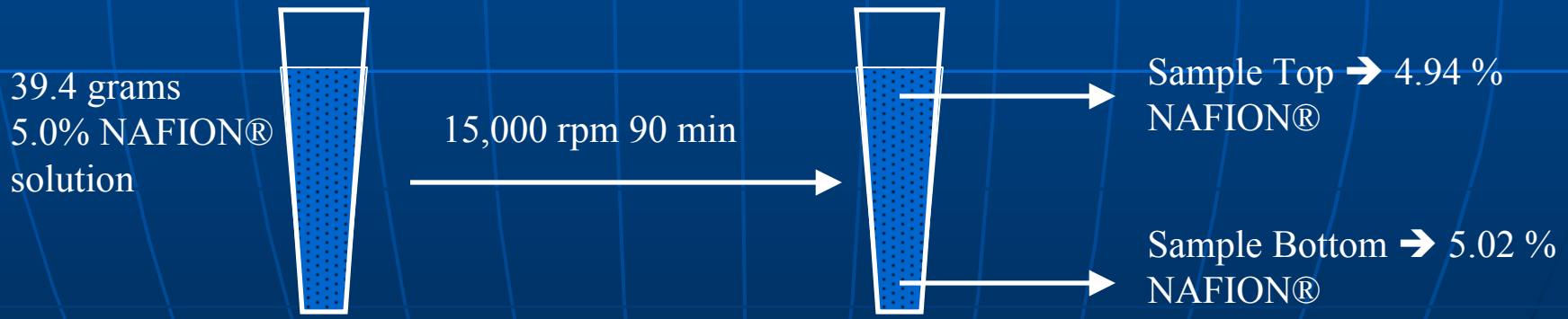


- Do NAFION™ particles settle upon Centrifugation?
  - This would limit effectiveness of Centrifugation as a separation process since NAFION® would settle with Pt

H-Type NAFION® solution, Centrifugation Experiment



L-Type NAFION® solution, Centrifugation Experiment



- Conclusion ➔ Ion Power NAFION® solutions do not separate upon Centrifugation

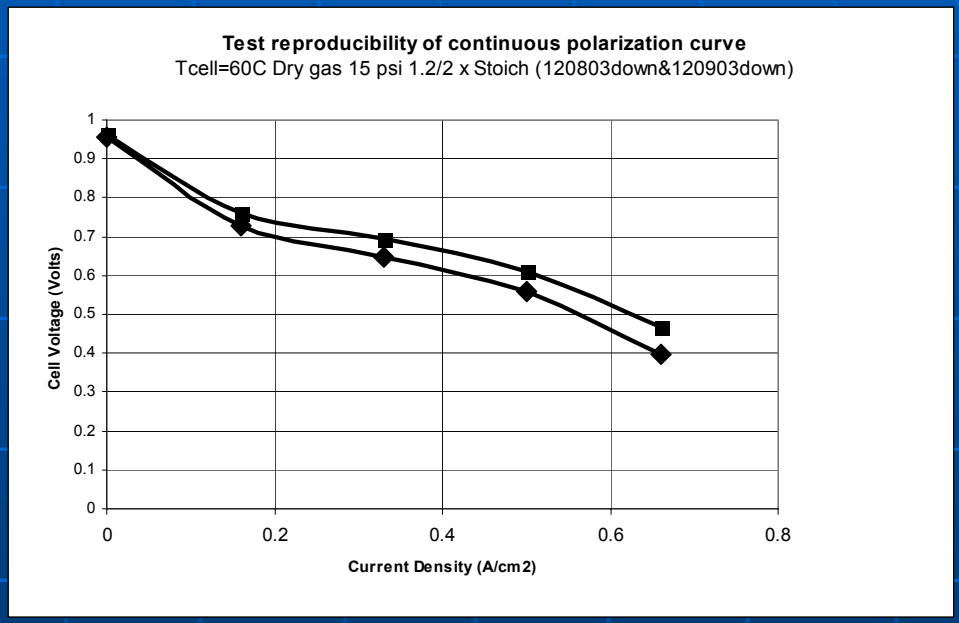
TASK 4 Remanufacture/Test

-5 kW Commercial fuel cell Systems Identified to be Purchased and run to failure at local Hydrogen Distribution facility with Program MEAs

- -Stainless Stack
- -Graphite Stack

-5kW is right size for Process Equipment identified in TASK 2

-Small scale tests identify baseline



Simple test set-up for Fast Aging under Dry Gas Operation, BOL Reproducibility single cell

Interactions & Collaborations

- DuPont:
  - Dennis Kounz; Supply of MEA's new & used materials evaluations
- Delaware State University
  - Prof. Goudy; Materials Characterization Catalyst & NAFION® vitality

Future Work

- Remainder of FY 2004:
  - Demonstration of vitality of separated materials
    - establish baseline characterization tools to determine vitality of separated materials
    - Demonstrate the degree of separation that can be achieved
  - Develop ability to controllably aged CCMs
    - Identify partners that can provide equipment to controllably age multi kW quantities of CCMs at once.
- FY 2005-2008:
  - Remanufacture and Test
    - Demonstrate a full stack running on remanufactured CCMs
  - Fuel Processor catalysts
    - Look at state of the art challenges, and identify resolutions to the challenges
  - Economic analysis
    - Based on best known pilot scale method estimate scale required to become competitive and profitable over existing methods of PGM recycling.